Thermophysical property measurements at high-temperatures for power engineering and additive manufacturing processes

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Abstract

To address the needs for increasing efficiency in power conversion, stratified structures like thermal barrier coatings, are used to increase operation temperature. Also advanced material processing like 3D laser printing of metals and ceramics are based on a layer-to-layer process at high temperatures, resulting in non-homogeneous components. Both systems require more and more detailed investigation methods to characterise the material properties of the resulting structures and to optimize the relevant processes. To address the required needs in advanced material characterisation recently an attempt was started to develop a unique measurement set-up for advanced material characterisation. This method is based on the well know laser flash principle, which was improved by adding supplementary heating sources and additional detection channels. Combining different heating mechanism and heating times with the two-dimensional measuring of the thermal flow across the sample enables the determination of different opto-thermal parameters and other material properties, e.g. mechanical contact, electrical conductivity or optical data, which also depend on or affect the flow of heat. In this paper we describe the implementation of the different optical methods to measure the thermal heat flow by point-like and two-dimensional temperature measurement and present first results on several samples.

1. Introduction

The availability of modern fabrication methods and improvements in material science allow the application of layered structures on metallic substrates to prevent them from harsh environments. A typical example is the application of thermal barrier coatings (TBC) on turbine blades to prevent the nickel-based substrate from being destroyed by the combustion gas. Using such TBCs allows the increase of the operation temperature of the gas turbine, which increases the efficiency of the power conversion process. Using Yttrium stabilized Zirconium-oxide layers on Nickel based substrates is state of the art for modern gas turbines. However, the adhesion of such layers on the metallic substrate is not sufficient and intermediate layers have to be used to optimize the mechanical contact. However, insufficient adhesion of TBCs is still one of the main problems in the area of power conversion gas turbines, which requires a frequent control and replacement of the turbine blades. Up to now, the bad adhesion usually can only be measured destructively by cutting of such systems and looking at the resulting cross-section by raster electron microscopes. These methods are quite accurate but destructive and can only be applied after the operation of such systems and not during their operation. In particular for stationary gas turbines, which operation is quite costly, the shutdown of these systems should be put to an absolute minimum.

Also components fabricated by the new additive manufacturing methods, which are based on a layer-on-layer process, are facing similar questions. The fancy and fragile structures built by such additive manufacturing methods pose the change of replacing conventionally fabricated components. However, only if they have the same material properties as the bulk material or structures manufactured by classical methods, like grinding, turning or milling. But is it not yet clear and guaranteed whether the novel processing technology is critically affecting the overall material properties of the component or not. Therefore, also in this area non-destructive testing of material properties, at best during the manufacturing process is highly demanded, in particular at the high fabrication temperature.

The recently presented approach [1] is based on the well-known laser-flash-method. Here the temperature evolution of a short heat pulse at the front side of the sample is monitored by a spot-like temperature measurement at the backside of the sample [2]. The time evolution of the backside temperature rise is affected by the thermal properties of the sample and can be used to determine detailed information about the thermal and structural constitution of the sample [3]. In the recently presented approach the laser-flash-method was extend to additional heating and detection features, also allowing heating and detection at front and back-side, either separately or simultaneously as well as with different time and spatial resolutions [1]. Here we present the newly introduced detection channels, allowing additional measurement of the front side temperature rise and also two-dimensional temperature measurements using thermal imagers. In chapter 2 the underlying theoretical principles are shortly presented and the innovative idea of the measurement principle is described.

Chapter 3 gives a survey of the experimental realizations while chapter 4 presents the implementation of the front side temperature detection system. In chapter 5 first results of front – and back-side temperature measurements are presented and an application to a special approach in the field of thermal barrier coating with the respective results is shown. The paper closes with a short discussion and an outlook to further application of the innovative method.

2. Theory and Method

In this section the basic theory and the fundamental laser flash method is presented and the ideas for the improvement yielding the new method are introduced.

2.1. Theory

For the investigation of the layered and in-homogeneous structures the laser flash method was applied. Here a thin sample is heated at the front side by a laser pulse and the back side temperature is recorded as shown schematically in Figure 1 [1].

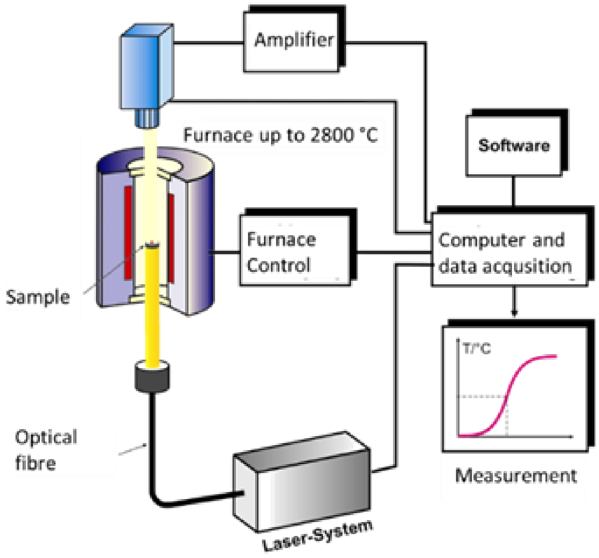

Figure 1: Schematic of the laser flash method [1]

Using a solution of the well-known one-dimensional heat-flow equation

$$\rho c_p \frac{\partial \Delta T}{\partial t} - \lambda \frac{\partial^2 \Delta T}{\partial x^2} = Q(t), \quad (1)$$

where ΔT is the temperature rise of the sample, $Q(t,x)$ the introduced heat power by the laser beam, λ the thermal conductivity, ρ the density and c_p the specific heat at constant pressure of the material under investigation. For an instantaneous heat pulse at the front side of a homogeneous sample the solution of Eq. 1 was already given in 1962 by Parker et al. [2]. Also for layered samples the result was already presented in literature long ago [e.g. 3]. Measuring and evaluating the temperature versus time curve and knowing the thickness of the sample the thermal diffusivity of bulk samples can be determined [2]. The thermal parameters of layered samples can, in principle, also be determined, as long as the other material properties and the properties of the substrate are known [3]. In the case of a two layered sample the contact resistance in-between the two layers can be determined and can be used as an indication of the adhesion of the two-layers [3].

2.2. New Method

Usually the sample is heated at one side (front side) and the temperature increase is measured at the opposite side (back side) [see eg. 2, 3].The intention of the new method is to determine additional material parameters and to obtain an indication for the adhesion without knowing the other material properties of the structure. To obtain these additional information, the laser flash method has to be improved significantly, in particular by adding additional heating and detecting channels. There have already been other attempts to adopt the laser flash method to measure other opto-thermal properties, mainly the hemispherical emissivity [see e.g. 4].

Here further improvements of the laser flash method are presented to enable the measurement of other optical and thermophysical properties. In particular the mechanical adhesion of layers is aimed at to develop a non-destructive approach for qualifying thermal barrier coatings. This improvement is reached by implementing additional sensor systems to measure additionally the front side temperature as well as implementing rear side heating of the sample. To facilitate the measuring of the lateral heat spread also imaging systems will be applied. The resulting improvement of the laser flash set-up is shown schematically in Figure 2. Applying the front- and back-side excitation and detection systems simultaneously will allow the measurement of additional thermophysical parameters, as due to the different schemes the various parameters will influence the resulting front- or back-side temperature rise in different ways and can, therefore, be measured more easily. To obtain depth sensitive information different wavelengths for the heating and measuring channel will also be used. At first a qualitative information of the thermal resistance as an indication for the mechanical adhesion is aimed at [5]. A good adhesion will usually be accompanied by a good thermal contact [5] and, therefore, in case of front side heating of such a good adhering structure will result in a fast decrease of the front side temperature and a good and instantaneous rise of the temperature at the backside. A bad adhesion is correlated to a high thermal resistance, which results in a high and slow decreasing front temperature and a slow and delayed increase of the back side temperature. This behaviour is graphically displayed in Figure 3.

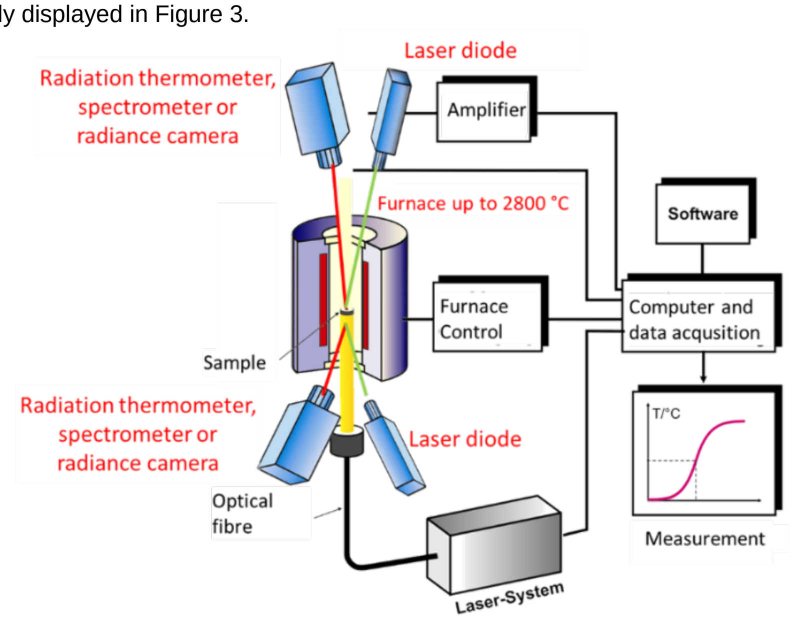

Figure 2: Improvement of the laser flash method [1]

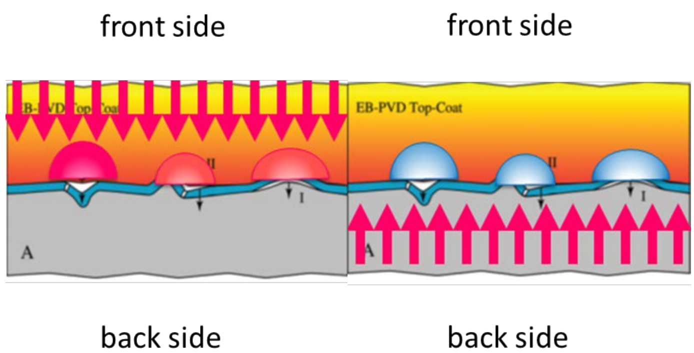

Figure 3: Idea of simultaneous front- (left) and back- (right) side heating and detection (picture adopted to [6], detailed explanation in text)

3. Experimental set-up

To measure the front side temperature rise an additional optical temperature measurement channel has to be used. For that a HEITRONICS KT15II was used. This radiation thermometer has a pyroelectric detector and, therefore, requires a chopper. Due to that reason the time constant of this radiation thermometer is restricted. However, the shortest time resolution of the KT15II is 5 ms and this time resolution is sufficient to monitor the time evolution of the front side temperature rise for most of the technological relevant material combination, such as metals, ceramics and compound materials used in additive manufacturing and in energy or process technology. However, as the time constant is decreasing the noise will increase and, therefore, this radiation thermometer can only be used for measuring the fast changing front-side temperature at sample temperatures higher than about 300 °C.

The KT15II is fixed at the bottom of the used laser flash set-up as shown in Figure 4. For the use as a front side laser flash detection system the KT15II must be directed to face the centre of the sample. The positioning can be performed with a pilot laser and a semi-transparent disc positioned in the sample position.

Figure 4: Position of the radiation thermometer KT15II

4. Results

At first the newly implemented laser flash set-up was validated by measuring the poco graphite and steel reference samples provided by the manufacturer of the laser flash set-up, Netzsch. The results of these measurements are shown in **Figure 5** and **Figure 6**. It can be seen that the obtained results are in excellent agreement with the reference values, indicating a validation of the set-up over the whole temperature range from room temperature up to 2800 °C.

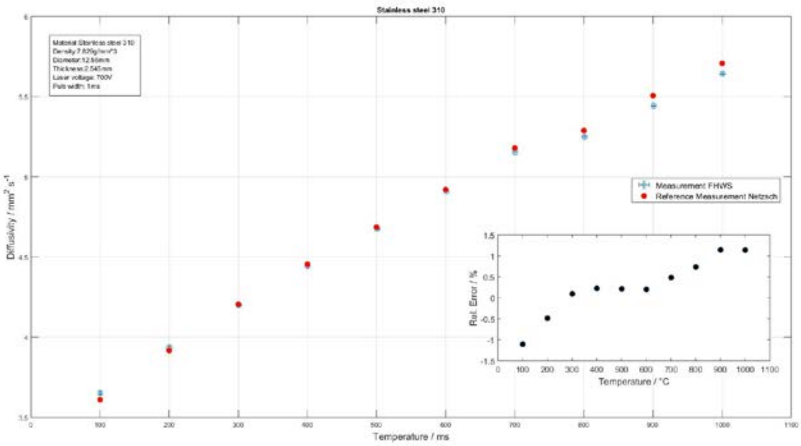

Figure 5: Results of the measurement of the steel reference sample. The Inset shows the relative deviation, which is well below 1.5% for nearly the whole temperature range.

In a second step the front side detection system was used to measure the front side temperature evolution after heating the front side of the sample by the laser pulse. For that measurements a sample mount was used, which allows the control of the heater temperature by a thermocouple and the radiation thermometer was focused on the centre of the heated sample area (see ***Figure 7***). This allows the measurement of the front side temperature rise simultaneously to the measurement of the back side temperature rise. The obtained measured general temperatures at the front side is shown in **Figure *8***. Here a series of heating by laser pulses is shown after the temperature of the furnace was stabilized to about 325 °C.

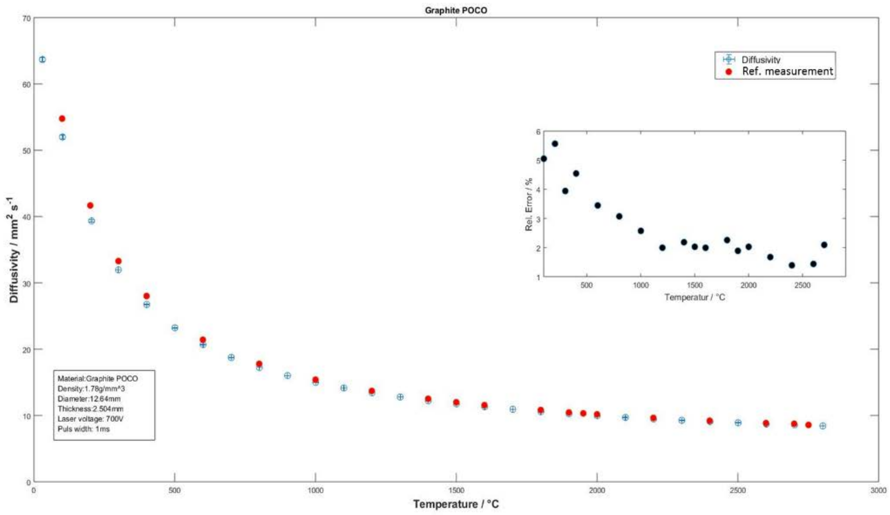

Figure 6: Results of the measurement of the poco graphite reference sample. The Inset shows the relative deviation, which is well below 6% for nearly the whole temperature range.

The signals of **Figure *8*** for a single pulse where shown in **Figure *9*** together with the simultaneously measured back-side temperature rise. The back-side signal was scaled to fit the front-side signal at longer times.

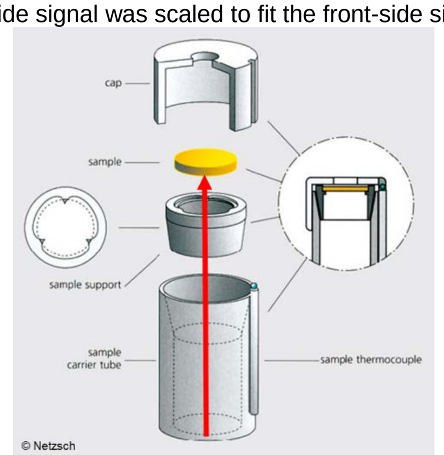

Figure 7: Sample mount and location of measuring spot of the radiation thermometer for measuring the front side temperature rise (red arrow at center of sample)

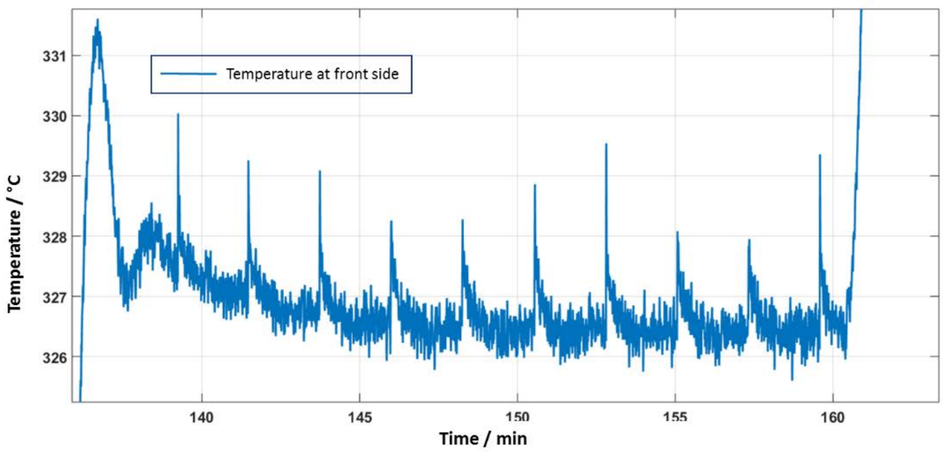

Figure 8: Result of the temperature measurement at the front side of the sample configuration of **Figure 7** using a long wavelength radiation thermometer.

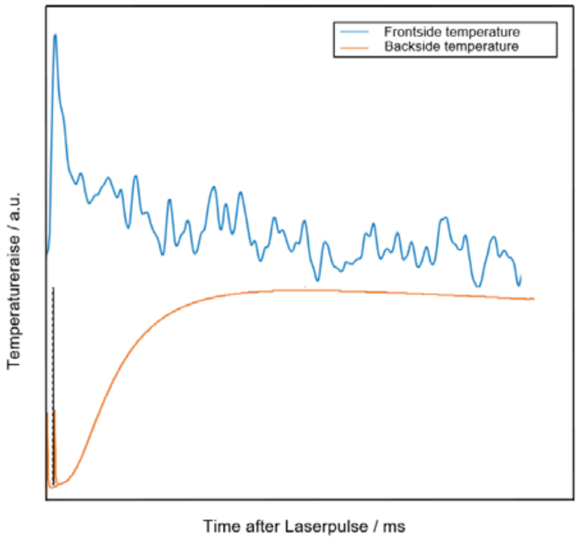

Figure 9: Simultaneously measured front- and back-side temperature. The back-side temperature rise was scaled to fit the front-side temperature rise after the first steep decrease.

5. Outlook and Acknowledgement

The results of **Figure *9*** show that the front side temperature rise can indeed be measured by the implemented radiation thermometer. In a next step the front and back side temperature measurement will both be performed with calibrated radiation thermometers to obtain the absolute temperatures at front and back side simultaneously. In a further step the tube furnace will be removed by a small size induction heater, which will then allow to use CCD, CMOS and thermal imager systems to investigate the lateral heat spread with high temporal and lateral resolution.

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Note: References to commercial products are provided for identification purposes only and constitute neither endorsement nor representation that the item identified is the best available for the stated purpose.

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